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Absence of phase transformation of dense anthracene from Raman scattering

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The high pressure vibrational properties of anthracene are investigated by using Raman scattering techniques in diamond anvil cells up to 7.1 GPa at room temperature. We present a detailed analysis of the normal vibrational modes of anthracene along with assignments of both symmetry and molecular motion. Our results demonstrate that almost all the modes shift toward higher frequencies and some peaks are broadened with increasing pressure. The smooth evolution of peaks corresponding to first and second nearest neighbor shell molecules with pressure rules out the possible existence of phase transformation. The mode Grüneisen parameters are obtained over the wide frequency range and relatively high pressure range.

Keywords: high pressure; aromatic hydrocarbons; Raman spectroscopy

1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) with π-electron networks have long attracted interest as potential materials for various electronic, optoelectronic, and optical applications.[1–7] PAHs have also been noted in astrophysics as candidates for interstellar media emitting unidentified infrared spectra.[8] Among PAHs, anthracene is certainly one of the most promising molecules showing prominent optical and electronic properties combined with high charge carrier mobilities.[9] Moreover, the strong anisotropy of the conductivity and the dielectric function were often found in crystals of short polycyclic organic molecules.[10,11] These are believed to be closely related to the specific way of molecular packing.

Pure anthracene is a rigid molecule which crystallizes at atmospheric pressure and room temperature in the monoclinic space group $P2_1/a$ with lattice constants of $a = 8.554$ Å, $b = 6.016$ Å, $c = 11.174$ Å, and $\beta = 124.60°$ and the long molecular axis points toward the crystal c direction.[12] The arrangement within the layers is called *herringbone* structure and is common for rigid rodlike aromatic hydrocarbons. The main feature of the arrangement of the anthracene molecule is that the long molecular axes of the two translationally inequivalent molecules are titled to each other. Crystal structure, molecular packing, and relative molecular orientation in

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*Corresponding author. Email: chen@hpstar.ac.cn a molecular crystal can be altered by change in temperature, pressure, electric, and magnetic fields.[13] Temperature-dependent single-crystal studies revealed some changes in the molecular packing of anthracene and weak changes in the conformation of the individual molecules.[14] Some of these experimental observations indicate the existence of a phase transition around 2.5 GPa while the other experiments do not support such a phase transition.[15] In one of the most recent works, the phase transition was observed at a pressure of 3 GPa using nitrogen as the transmitting medium.[16] However, high pressure, X-ray diffraction experiments [17] on anthracene suggested that there was no evidence of a structural phase transition up to 10 GPa at room temperature consistent with the report from [18]. First-principles calculations [19,20] on anthracene revealed isothermal bulk-moduli, electronic band structures and dielectric tensors as a function of the unit cell volume. Overall, the variation of the structural quantities such as cell parameters, volume and interaction energy with increasing pressure does not show any discontinuity under pressure. Clarification of the existence or nonexistence phase transition of anthracene is an important issue for better understanding its high pressure behaviors such as possible metallization [21] and even superconductivity.[22,23]

High pressure Raman spectroscopy is a very powerful tool to probe the changes in intermolecular interactions and dynamics in molecular solids without changing the chemical composition.[24,25] Changes in the vibrational spectrum can be used to probe chemical and physical changes associated with static high pressure.[26] In this work, we present the results of the high pressure Raman scattering spectra of anthracene up to 7.1 GPa at room temperature. The information of the pressure-related changes of molecular vibrations and the corresponding responses of the Grüneisen parameters are provided.

2. Experimental details

Anthracene of 98% purity, colorless crystal, was purchased from TCI Co. and used without any further purification. High pressure Raman measurements were carried out in symmetric diamond anvil cells (DACs) with the culets of 300 μm. A 100 μm hole which was drilled in the center of stainless-steel gasket served as the sample chamber. To calibrate the pressure a small ruby chip was placed in the sample chamber with the samples whereby measuring the wavelength shift of the ruby R_1 fluorescence line gave an accurate measure of applied pressure.[27]

In order to guarantee hydrostatic pressure during the experiments a 4:1 methanol–ethanol mixture was used as the pressure transmitting medium. In-house Raman system with a spectrometer (with 1800 lines/mm grating) was used for the measurements, giving a resolution of 1 cm^{-1} and the lowest wavenumber till 10 cm^{-1}. The Raman spectra were measured in backscattering geometry with visible laser excitation (659.8 nm) with power less than 50 mW. The spectra were collected from 10 to 1800 cm^{-1}. All the spectra were measured at room temperature.

3. Results and discussion

3.1. *High pressure vibrational properties*

Raman scattering, which measures phonons (lattice and molecular vibrations) in Brillouin zone center, possesses a very high selectivity and is known to be a powerful technique for the investigation of both chemical reactivity and even subtle structural distortion both within a space group (via band shifts) and due to phase transitions (via band splitting and/or soft modes). The anthracene molecule contains 24 atoms, its spectrum possesses 66 intramolecular modes, including 45 in-plane modes (12A$_g$, 11B$_{3g}$, 11B$_{1u}$, 11B$_{2u}$) and 21 out-of-plane modes (4B$_{1g}$, 6B$_{2g}$, 5A$_u$,

$6B_{3u}$).[28] Eight of these eignvectors at 115, 388, 743, 957, 996, 1177, 1316, and 1471 cm^{-1} are shown in Figure 1. Most of the phonon branches of solid anthracene (not shown) have very little dispersion, which reflects the molecular nature of the solid. The modes below 500 cm^{-1} are due to skeletal deform vibrations involving both C and H atoms that cause only small changes in the bond length between neighboring atoms. The vibrations between 700 and 1300 cm^{-1} are mostly

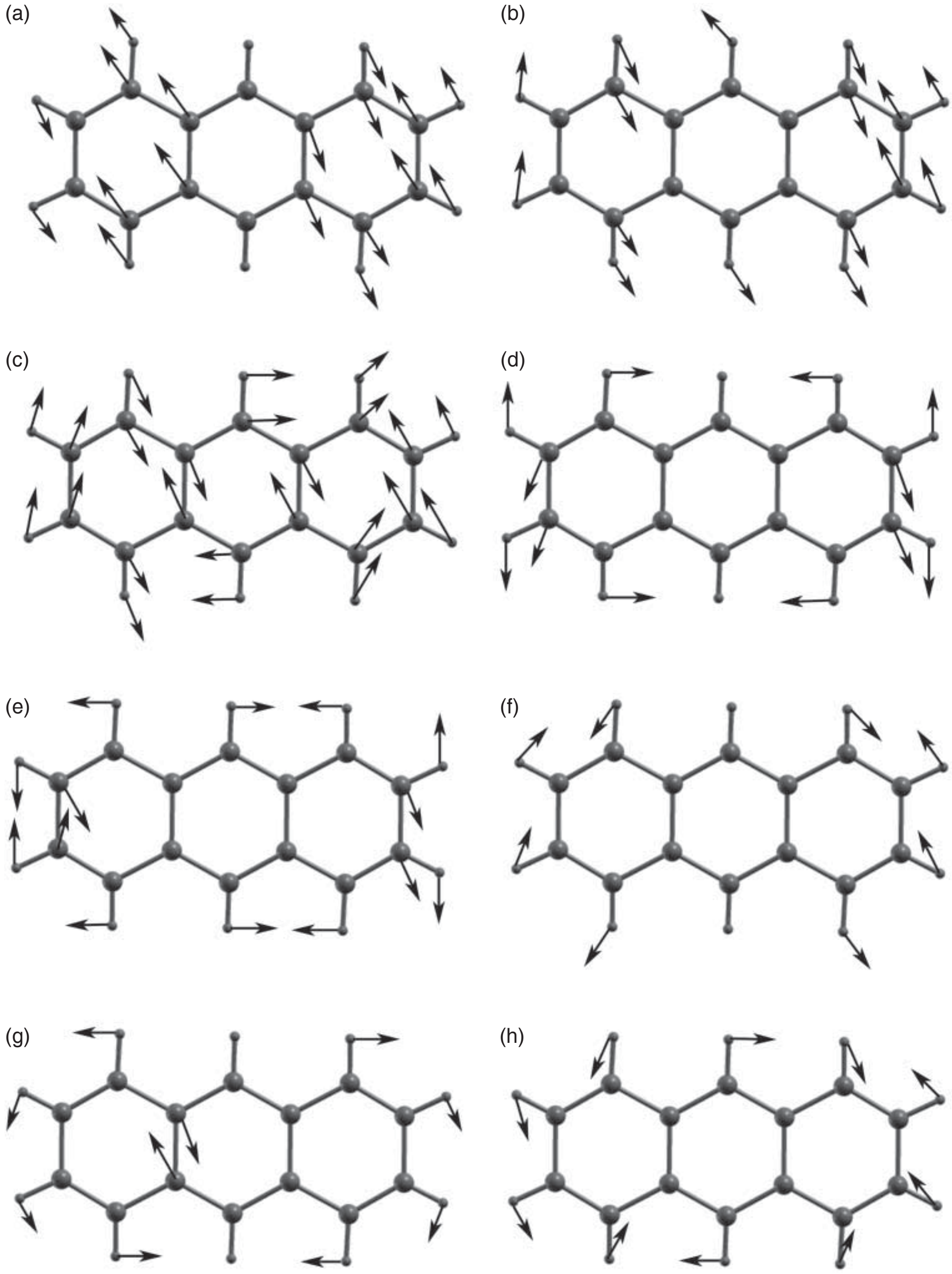

Figure 1. Selected vibrational eigenmodes of anthracene: (a) $\mathcal{V}_2$ at 115 cm^{-1}, (b) $\mathcal{V}_7$ at 388 cm^{-1}, (c) $\mathcal{V}_{11}$ at 743 cm^{-1}, (d) $\mathcal{V}_{14}$ at 957 cm^{-1}, (e) $\mathcal{V}_{15}$ at 996 cm^{-1}, (f) $\mathcal{V}_{17}$ at 1177 cm^{-1}, (g) $\mathcal{V}_{19}$ at 1316 cm^{-1}, (h) $\mathcal{V}_{22}$ at 1471 cm^{-1}.

in plane, while the modes between 1300 and 1600 cm^{-1} have C–H bending and C–C bending and stretching characters. Two typical examples of such modes are shown in Figure 1(f) and 1(g), with frequencies of 1177 and 1316 cm^{-1}, respectively.

According to the selection rule, the A_u, A_g, B_{1g}, B_{2g}, and B_{3g} vibrations are Raman active. The type of vibrations was well assigned to each Raman signal of anthracene at ambient conditions and is summarized in Table 1. Within our work, some low-intensity modes, such as CH stretching modes located between 3000 and 3200 cm^{-1} could not be detected. Our data at ambient pressure are in good agreement with the previous results.[29] The similar vibrational properties have been investigated for picene [30], phenanthrene [31], and coronene.[32]

The Raman spectra obtained for anthracene at various pressures up to 7.1 GPa in the region from 10 to 1800 cm^{-1} at room temperature are shown in Figure 2. It should be noted that the intensities of the vibrational modes drastically decrease when pressure is increased up to 6.8 GPa, accompanied with the increasing fluorescence background. With increasing pressure up to 7.1 GPa, some of peaks nearly vanish except for the 61, 743, 1392, and 1547 cm^{-1} that could be observed. The Raman spectra above 7.1 GPa were significantly obscured by an increase in the fluorescence background, measurements beyond this pressure were not pursued. This similar behavior was often observed in aromatic compounds such as benzene,[33] pyrene,[34] and picene.[30]

For organic molecules, the lattice and intramolecular vibrational modes are situated in different regions of frequency.[35] The lattice modes, such as translations representing the motions of rigid molecules under whole molecular interaction, are in the low-frequency region of the Raman spectra, while the intramolecular modes in the high-frequency region such as bond stretches and rotations representing the vibration of atoms in a molecule. For anthracene, the lattice modes below 200 cm^{-1} at ambient pressure are external phonon modes, and the corresponding modes

Table 1. Raman modes and their assignments, the observed frequencies and the Grüneisen parameter of the corresponding mode at ambient conditions from Ref. [29] for anthracene.

Raman mode	Assignment	Obs. (cm^{-1})	Grüneisen parameter γ_i
L_1	Lattice mode	28	2.367
L_2	Lattice mode	34	2.241
ν_1	Skeletal deform	61	1.346
ν_2	Skeletal deform	115	0.867
ν_3	Skeletal deform	144	0.705
ν_4	Skeletal deform	150	0.770
ν_5	Skeletal deform	245	0.169
ν_6	Skeletal deform	291	–
ν_7	Skeletal deform	388	0.074
ν_8	Skeletal deform	467	0.017
ν_9	Skeletal deform	510	0.014
ν_{10}	C–H out-of-plane bending	728	–
ν_{11}	Skeletal deform	743	0.027
ν_{12}	C–H out-of-plane Bending	886	–
ν_{13}	Skeletal deform	907	–
ν_{14}	C–H out-of-plane bending	957	–
ν_{15}	C–H in-of-plane bending	996	0.021
ν_{16}	C–H in-of-plane bending	1154	0.009
ν_{17}	C–H in-of-plane bending	1177	0.015
ν_{18}	C–H in-of-plane bending	1246	0.031
ν_{19}	Ring stretching	1316	–
ν_{20}	Ring stretching	1392	0.023
ν_{21}	Ring stretching	1448	–
ν_{22}	Ring stretching	1471	0.013
ν_{23}	Ring stretching	1547	0.015
ν_{24}	Ring stretching	1634	0.017

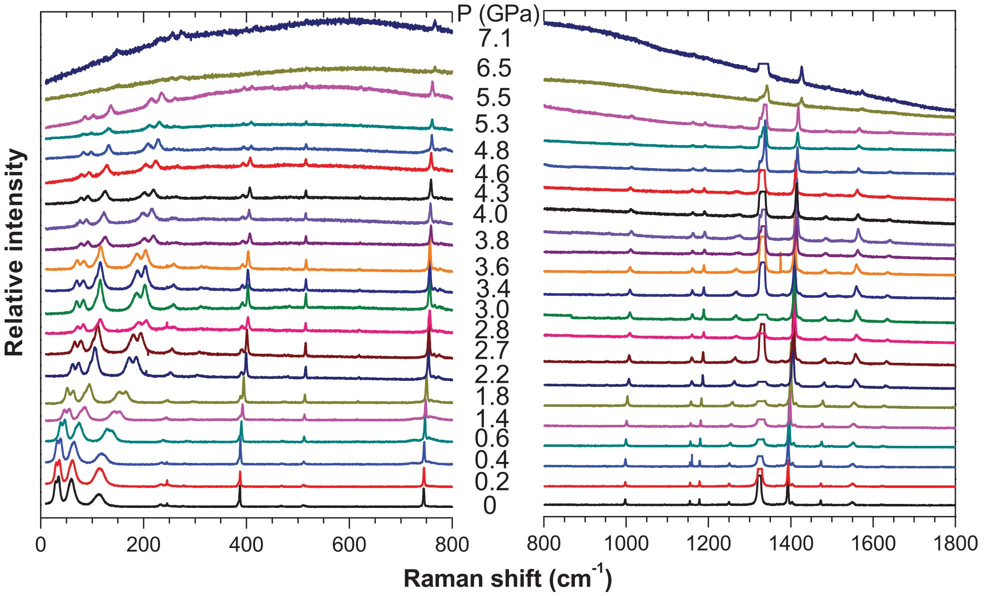

Figure 2. Raman spectra in the frequency range 10–1800 cm^{-1} at various pressures from 0 to 7.1 GPa. The peaks near 1330 cm^{-1} are due to the diamond anvils.

in perdeuterated anthracene have previously been studied up to a pressure of 0.8 GPa.[36] However, only four peaks at 28, 34, 60, and 115 cm^{-1} can be observed within this region at ambient pressure in our measurement. At 1.4 GPa, the peak at 115 cm^{-1} associated with lattice mode splits into two modes. Whilst the skeletal deform mode v_8 originally at 467 cm^{-1} disappears. More interestingly, the overlapping peaks with the frequency 245 cm^{-1} corresponding to skeletal deform combine into a single peak and loss of intensity above 4.3 GPa. Further increase of pressure up to 5.5 GPa shows a decrease in intensity associated with the lattice modes to the point where they are lost in the increasing fluorescence background.

For the higher frequencies significant changes can also be observed as pressure increases. The modes with frequencies 743, 1392, and 1547 cm^{-1} are weakened and broadened by rotations and molecular interactions (vibrational relaxation) with increasing pressure. Additionally, in frequencies ranging from 900 to 1600 cm^{-1}, no apparent discontinuity, disappearance, or splitting was observed in the C–C stretching vibration modes under pressure, implying the stability of carbon bonding below 6.8 GPa, again supporting the stability of the phase. Moreover, within our data the lattice modes exhibit more drastic changes than intramolecular modes, which are due to there being greater intermolecular distortions than intramolecular under applied pressure.

3.2. *Pressure-induced Raman frequency shifts and Grüneisen parameters*

In order to precisely determine the pressure effects on the crystal vibrations, the pressure dependencies of Raman modes are depicted in Figures 3 and 4. With increasing pressure, all modes shift gradually toward higher frequencies. Additionally, the pressure-induced shifts for the high frequencies are nearly linear with pressure, whereas the pressure-induced shifts in the low frequencies shown in Figure 3 are nonlinear. The initial slopes of the pressure-induced frequency shifts for the low frequencies are in the range 10–20 cm^{-1}/GPa. In contrast, the intramolecular vibrons above 200 cm^{-1} yield fairly linear pressure shifts in the range 1.2–6.8 cm^{-1}/GPa. There are two main pressure effects on the crystal vibrations: large shifts of the modes in the lattice modes region and slight modification of the intramolecular modes in the high-frequency region.

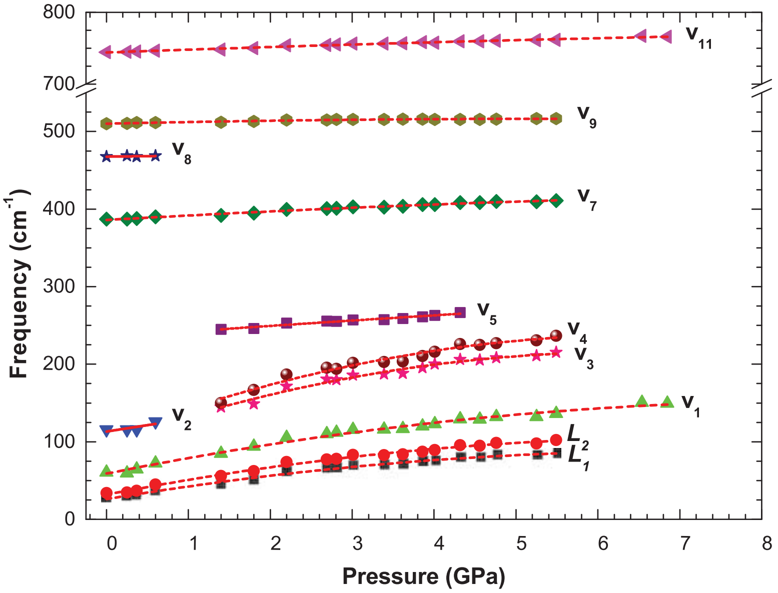

Figure 3. The pressure dependence of the vibrational frequencies of anthracene in the low-frequency range of 10–800 cm^{-1} at room temperature.

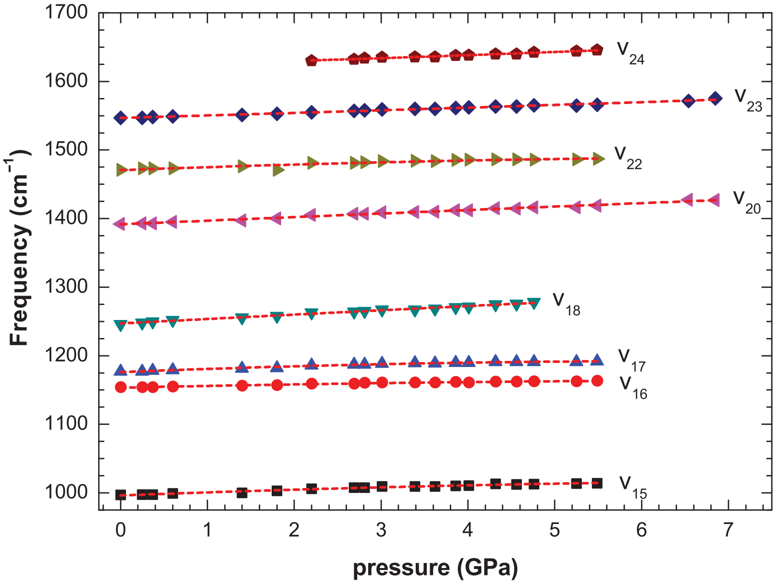

Figure 4. The pressure dependence of the vibrational frequencies of anthracene in the frequency range of 800–1800 cm^{-1} at room temperature.

To further understand the intermolecular interaction upon compression, we also calculate the mode Grüneisen parameters for representative Raman shifts of anthracene.

Using a standard definition for the mode Grüneisen parameter γ_i and with $\gamma_i = -(\mathrm{dln}(\upsilon_i)/\mathrm{dln}(V)) = -(V/\nu_i)(\mathrm{d}\nu_i/\mathrm{d}V) = B_T \cdot (1/\upsilon_i)(\mathrm{d}\upsilon_i/\mathrm{d}_p)$, where ν_P is the vibrational frequency at an applied pressure P, V_P is the volume of the bulk solid at pressure P, and $k_\mathrm{V} = (1/B_T) = -(1/\mathrm{V})(\mathrm{dV}/\mathrm{dP})$ is the volume compressibility and B_T is the bulk volume modulus. When taking $B_T = 6.08 \pm 0.24$ GPa from [17], we obtained the relative γ_i values by using our obtained values of υ_i and $\mathrm{d}\upsilon_i/\mathrm{dP}$.

The results are summarized in Table 1. There are discrepencies between our data and those in the literature.[16] As no calculation details were provided in the previous study, we suspect that the authors used a different bulk modulus. In the present work, the more reliable volume–pressure results were used in our calculations.[17] Grüneisen parameters indicate the vibration frequencies dependence of the volume, it can reflect the anharmonic effects, indicating how sensitive the vibrational eigenfrequencies are to the change of volume. A large Grüneisen parameter can be obtained based on the large change in frequency for a small variation in crystal volume. Here, we have collected data at a relatively high pressure range and more wide frequency range. The relatively large γ values observed for intermolecular phonon modes are due to the relatively large compression of the vibrational coordinate, while the relatively small values observed for intramolecular vibrons are due to the fact that compression occurs principally along intermolecular coordinates orthogonal to the vibron coordinate. These may indicate that the distance between the layers is more apt to be compressed.

Meanwhile, many other experimental investigations have been carried out for anthracene under pressure. X-ray diffraction measurements were reported up to a pressure 10.5 GPa.[37] Dreger et al.[38] reported the changes of the fluorescence spectra at pressure up to 5 GPa. According to our results, all Raman shifts have smooth pressure dependences. There is also no evidence of a structural phase transition up to 7.1 GPa at room temperature. Figure 5 shows the pressure dependence of the full-width half-maximum (FWHM) of selected vibrational modes. The FWHM of the peaks at $\mathcal{V}_2$ (115 cm^{-1}) and $\mathcal{V}_9$ (510 cm^{-1}) are observed to broaden abruptly at pressure near 2.2 and 2.8 GPa, respectively. Nevertheless, we do not observe any discontinuities in the Raman shifts under pressure, only changes in the FWHM as discussed above. One possible reason for the line width broadening in the high pressure experiments is the pressure gradient in the sample due to nonhydrostaticity of the medium at relatively high pressure. Thus, the molecules would experience slightly different pressures for the different locations within the pressure cell. As can be seen, the broadening and evolution with pressure differ among different modes. This implies that the distortions of different bonds of the molecules lead to the broadening of the linewidths. The absence of the phase transition of the studied system is consistent with other experimental observations by Oehzelt et al.[17] Our Raman scattering measurements offer direct evidence for the structural stability of anthracene at high pressures. This indicates that the

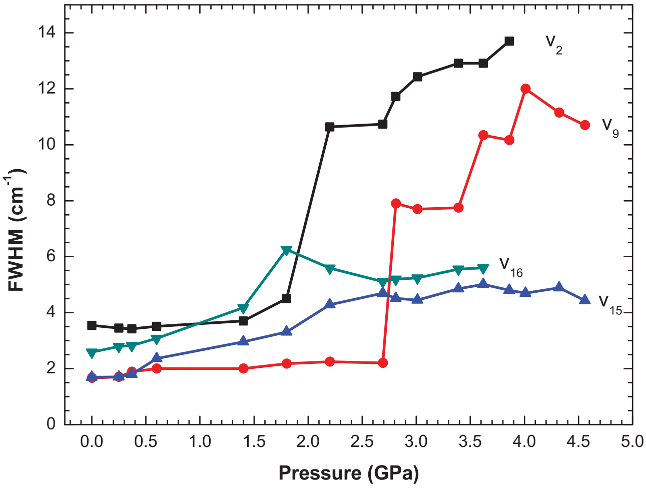

Figure 5. The pressure dependence of the FWHM of anthracene of selected vibrational modes of $\mathcal{V}_2$, $\mathcal{V}_9$, $\mathcal{V}_{15}$ and $\mathcal{V}_{16}$.

possible metallization of compressed anthracene could be a result of the band gap closure within the same structure feature.

4. Conclusions

We have investigated the vibrational properties of anthracene at high pressures up to 7.1 GPa by the Raman scattering technique in a DAC at room temperature. A complete characterization of Raman spectra within the region from 10 to 1800 cm^{-1} under pressure is given for this material. We have demonstrated that the vibrational frequencies of all the modes increase with increasing pressure. The loss of Raman signals above 6.8 GPa indicates the possible presence of the shortened distance between molecules at high pressures. Our data over a wide frequency range do not support the existence of the possible phase transition at 7.1 GPa as observed or reported previously, rather than the molecular deformation instead. Based on high-resolution Raman spectra, wide frequency range, and wide pressure range, we obtained the mode Grüneisen parameters of this material, which are useful for understanding the vibrational changes and intermolecular interactions.

Disclosure statement

No potential conflict of interest was reported by the authors.

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